Discussion. The structure of the title compound consists of discrete molecules separated by normal van der Waals distances. The Cu atom is coordinated to two bidentate ligands and has a distorted square-planar geometry with one Cu–O bond, two chemically equivalent Cu–N bonds which are not significantly different from each other in length and a Cu–N distance which is 0·04 Å longer than the other two Cu–N distances. The Cu–N distances observed in the title compound fall within the range observed for the corresponding distances reported in $[Cu(C_9H_{11}N_2)_2]$ (Wei, 1972a) and the other related complexes referred to in Wei's (1972a) paper. The angles around Cu also deviate from the values for square-planar angles, e.g. trans O–Cu–N and N–Cu–N angles are 169·5 (1) and 173·5 (1)°, respectively, and the cis angles are: O–Cu–N 91·5 (1), 82·2 (1), N–Cu–N 82·6 (1) and 103·8 (1)°. It is interesting to note that the shorter Cu–N bond distances which involve N(1) and N(3) are associated with the largest cis angle around Cu.

All the five-membered rings are individually planar to within 0·012 (4) Å. The short N(2)–C(5) bond length of 1·29 (4) Å is comparable with the values reported in the Schiff-base–metal complexes with Co^{II} [1·27 (1) Å (Wei, 1972a)], Ni^{II} [1·287 (4) Å (Braun & Lingafelter, 1966)] and Cu^{II} [1·25 (1) Å (Cheeseman, Hall & Waters, 1966)].

The bond distances and angles in the ligand agree well with those reported for similar complexes, e.g.

$[Zn(C_9H_{15}N_2)_2]$ (Kanters et al., 1983), $[Co(C_9H_{15}N_2)_2]$ (Wei, 1972a), $[Ni(C_9H_{15}N_2)_2]$ (Wei & Einstein, 1972) and $[Cu(C_9H_{15}N_2)_2]$ (Wei, 1972b).

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Structure of Bis(2,2',2''-triaminotriethylamine)lead(II) Dichloride

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Abstract. $[Pb\{N(CH_2CH_2NH_2)_3\}_2]Cl_2$, $M_r = 570·6$, trigonal, $R\bar{3}$, $a = 8·1556$ (8), $c = 26·198$ (4) Å, hexagonal setting, $U = 1509·1$ (3) Å^3, $Z = 3$, 1·808(1,4-dibromobutane) $< D_m <$ 1·95(iodoethane), $D_x = 1·883$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0·71069$ Å, $\mu = 8·73$ mm^{-1}, $F(000) = 840$, $T = 298$ K, $R = 0·036$ and $wR = 0·042$ for 1093 unique reflections. The compound is crystallized from a commercial grade of triethylenetetramine solution. The Pb atom at a symmetry center is surrounded by six terminal N atoms of the ligand with Pb–N bond distance of 2·742 (6) Å, and two tertiary N atoms on the crystallographic threefold axis at 2·829 (6) Å. The coordination polyhedron around the Pb atom is a distorted cube contracted around the threefold axis.

Introduction. Commercially available triethylenetetramine (trien) has been found to contain more than 10% of 2,2',2''-triaminotriethylamine (tren) (Utsuno, Yoshikawa & Tahata, 1985). It is a nuisance for the coordination chemist to separate tren from trien before using trien.

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In the course of our study on the adduct formation of lead(II) halides with Lewis bases, we accidentally found that the tren molecule selectively bonds to lead(II) chloride ($PbCl_2$) in EtOH solution and gives beautiful rhombohedral crystals of formula $[Pb(tren)_2]Cl_2$. This paper deals with the eight-coordination geometry around the Pb atom of the selectively formed title compound.

Experimental. $PbCl_2$ (2 mmol) was dissolved in 20 ml of commercial-grade trien solution. After a week or so, colorless brilliant rhombohedral crystals had grown in the solution by standing at room temperature.

Crystal dimensions ca $0.2 \times 0.2 \times 0.2$ mm, 1093 independent reflections with $2\theta < 60°$ (h $-11\rightarrow11$, k $0\rightarrow11$, l $0\rightarrow36$) were measured, Rigaku AFC-5 diffractometer, graphite-monochromated Mo $K\alpha$, θ–2θ scan mode, scan range $(1 + \frac{1}{3}\tan\theta)°$. Unit-cell dimensions refined from least-squares fit of 25 2θ values of independent reflections ($29 < 2\theta < 34°$). Corrections applied for Lorentz and polarization factors, absorption and extinction ignored. All of the intensities have $|F| > 3\sigma(|F|)$ and were used for structure determination. Solved by heavy-atom method and refined by block-diagonal least squares to final R $= 0.036$ and $wR = 0.042$. $\sum w |F_o^2 - F_c^2|/\sum w |F_o|^2$ minimized, $w = [\sigma^2(F_o) + 0.02F_o^2]$. H atoms located from geometrical calculation and refined assuming isotropic thermal parameters. $S = 3.1378$, $(\Delta/\sigma)_{max} <$ 0.5 for non-H atoms, max.$\Delta\rho = 3.11$ e Å^{-3} around Pb. Complex neutral atomic scattering factors (*International Tables for X-ray Crystallography*, 1974). *UNICS*-III program system (Sakurai & Kobayashi, 1979) on a FACOM M-360 at Computer Center of Josai University.

The final atomic coordinates with e.s.d.'s for non-H atoms are listed in Table 1* and the atomic numbering scheme is given in Fig. 1.

Discussion. The structure of the $[Pb(tren)_2]^{2+}$ cation is illustrated in Fig. 1. The bond distances and angles are in Table 2.

The Pb atom at the center of symmetry is surrounded by eight N atoms in a distorted cube which is contracted around the threefold axis. The $[Pb(tren)_2]^{2+}$ ion has S_6 symmetry. The bond distance Pb—N(1) is longer than Pb—N(2). However, these are longer than the bond lengths observed in the hexacoordinated system of a 2.54–2.64 Å $[PbI_4(C_5H_5N)_2]$ octahedron (Miyamae, Toriyama, Abe, Hihara & Nagata, 1984). The N···N distances within the edges of the coordi-

Table 1. *Atomic parameters*

Positional parameters are multiplied by 10^4. Thermal parameters are given by the equivalent isotropic temperature factors (Å^2).

	x	y	z	B_{eq}
Pb(1)	0	0	0	1.57 (1)
Cl(1)	0	0	−2767 (1)	3.00 (5)
N(1)	0	0	−1080 (2)	1.72 (13)
N(2)	−3424 (7)	−963 (7)	−434 (2)	2.91 (16)
C(1)	−1862 (7)	−348 (7)	−1260 (2)	2.29 (16)
C(2)	−3501 (7)	−1764 (8)	−936 (2)	2.65 (16)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s within the* $[Pb(tren)_2]^{2+}$ *cation*

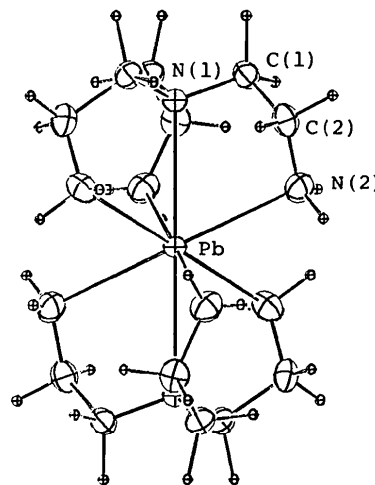|			
Pb—N(1)	2.829 (6)	N(1)—Pb—N(2)	65.5 (1)
Pb—N(2)	2.742 (6)	N(2)—Pb—N(2^i)	76.0 (2)
N(1)—C(1)	1.475 (6)	N(2)—Pb—N(2^{ii})	104.0 (2)
C(1)—C(2)	1.515 (7)	Pb—N(1)—C(1)	108.6 (3)
C(2)—N(2)	1.455 (8)	Pb—N(2)—C(2)	108.2 (4)
		N(1)—C(1)—C(2)	113.5 (5)
		N(2)—C(2)—C(1)	109.8 (4)
		C(1)—N(1)—C(1^{ii})	110.3 (4)

Symmetry operations: (i) $-y, x+y, -z$; (ii) $y, -x-y, z$.

Fig. 1. Structure of $[Pb(tren)_2]^{2+}$ with atom numbering. Thermal ellipsoids are at 50% probability level (Johnson, 1976).

nation polyhedron are 3.013 (6) and 3.377 (7) Å for N(1)···N(2) (within a ligand) and N(2)···N(2^i) [between the adjacent ligands; symmetry operation: (i) $-y, x+y, -z$], respectively. Thus the coordination cube is slightly elongated along the threefold axis.

A perspective view of the crystal is shown in Fig. 2. The Cl$^-$···$[Pb(tren)_2]^{2+}$···Cl$^-$ group is on the crystallographic threefold axis. The structure could be depicted so that the $[Pb(tren)_2]^{2+}$ cations stack in hexagonal closest packing and the Cl$^-$ anions are located on every tetrahedral site. In this situation, the Cl$^-$ ion forms three Cl···H—N(2) hydrogen bonds [Cl···N(2^{ii})] distance 3.534 (5) Å; symmetry operation: (ii) $\frac{2}{3}+x, \frac{1}{3}+y, -\frac{1}{3}+z$].
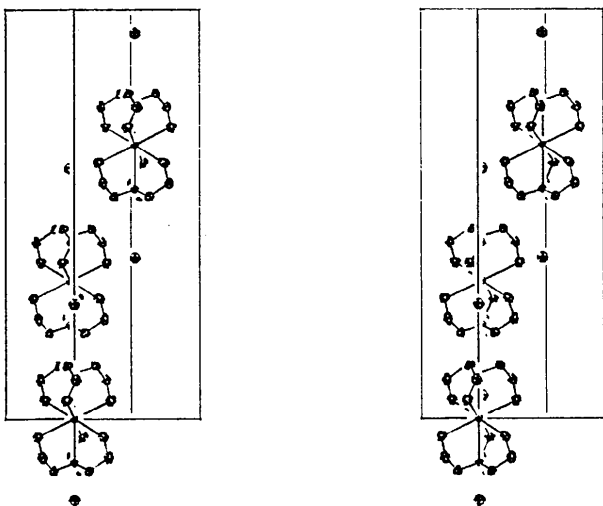
Fig. 2. Packing view of the crystal. View direction along −b*, a horizontal, c vertical.

The compact crystal packing described above and the tight fit between the Pb atom and tren molecule could be the reason why the title compound is easily separated from the trien solution. On the other hand, isomorphous compounds have not been crystallized from the reaction of trien solution with $PbBr_2$ or PbI_2, because of expectations of size differences in the anions.

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The Structure of an Intensely Coloured Fe^II Complex, Diaquabis(2-nitro-1,3-indandionato)iron(II) Dihydrate

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Abstract. $[Fe(C_9H_4NO_4)_2(H_2O)_2].2H_2O$, $M_r = 508·18$, monoclinic, $P2_1/c$, $a = 8·763$ (3), $b = 15·006$ (5), $c = 15·680$ (5) Å, $\beta = 105·56$ (2)°, $V = 1986$ (3) Å^3, $Z = 4$, $D_m = 1·72$ (1), $D_x = 1·699$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0·71073$ Å, $\mu = 8·36$ cm^{-1}, $F(000) = 1040$, $T = 295$ K, $R = 0·040$ for 3579 observed reflections. The Fe^II ions are placed on a general position and coordinated to six oxygen atoms in a slightly distorted octahedral configuration. Each 'nitronate' ligand has two different C—O and N—O bond distances. The cause of the dark colour of the title compound is discussed. A Mössbauer spectrum of the title compound has been recorded.

Introduction. During work with 2-nitro-1,3-indandione (Simonsen & Jacobsen, 1977) we became aware of the title coordination compound (*A*), first described by Wanag (1936). The structure of (*A*) was determined as part of a study of nitro compounds and their salts and especially to investigate the 2-nitro-1,3-indandionato ion as a ligand and to look for a structural explanation of the deep purple colour of (*A*). The colour of (*A*) is so characteristic that 2-nitro-1,3-indandionato has been suggested as a reagent for Fe^II (Wanag, 1936).

Experimental. The crystals used in the structure determination were prepared by allowing aqueous solutions of $(NH_4)_2Fe^{II}(SO_4)_2$ and 2-nitro-1,3-indandione to diffuse slowly into each other in the horizontal part of an H-shaped tube. D_m by flotation in a mixture of CCl_4 and CH_2BrCl. Crystal 0·36 × 0·13 × 0·08 mm. The crystals are black, with a violet lustre. Thin crystals are red-violet in transmitted light. Diffractometer: Enraf–Nonius CAD-4F, graphite-monochromatized Mo $K\alpha$ radiation, lattice parameters from Guinier–Hägg photographs with Cu $K\alpha_1$ ($\lambda = 1·54051$ Å), 19 reflections with $5·23 < \theta < 16·68°$, Si used as an internal standard. 5750 unique reflections (h 0→12, k 0→21, l −21→20) with $2·0 < \theta < 30·0°$, 3579 with $I > 2·5\sigma(I)$ used in refinement process together with 'less-than' reflections [$I < 2·5\sigma(I)$] with calculated values greater than the observed (5034 contributing reflections). $\omega/2\theta$-scan technique, scan angle $\Delta\omega = (1·00 + 0·35\tan\theta)°$. Standard reflections 35$\bar{4}$, 32$\bar{7}$, 361 and $\bar{2}$1$\bar{6}$ used for orientation control every 100 reflections, 353 used for intensity check every third hour of exposure time, standard intensity variations <3·0% of mean value. Lp correction, absorption